International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.11 No.01, pp 108-115, 2018

Tricalcium Phosphate Composites for Orthopedic applications: Preperation and Characterization

A.J.R. Nelson[1]*, M. Kavitha[2]and K.S. Vinoth[3]

[1]Department of Mechanical Engineering, Karpagam University, Coimbatore-641 021, Tamilnadu, India.
[2,3]Department of Metallurgical Engineering, PSG College of Technology, Coimbatore-641 004,Tamilnadu, India.

Abstract : In the current scenario, the requirement for biomaterials is increasing since repair and rejuvenation of the damaged/injured human body parts are increasing due to enhanced life period and medication. In this study composite materials made up of TriCalcium Phosphate(TCP) with the reinforcement of polyglactin and Catgut fibers were produced and characterized. Slip casting route was used to synthesizeTCP composites with2.5 wt% polyglactin and 2.5 wt% catgut fibers along with two more composite samples each containing 5 wt% of polyglactin and catgut reinforcements respectively. Sodium silicate acted as a very good binder thus increasing its tensile strength upto 29 MPa. Drilling on the composites was done to study the machinability and drilling characteristics of the composite to have screws/bolts, when used as a bio implants. In order to ensure bio compatibility of the composite, an artificial body fluid test was done and observed that weight of the samples increased after two days of dipping. Above results showed that this prepared composite may be a viable bio implant material with sufficient strength and hardness with good biocompatibility.
Keywords : Biomaterial, Tri Calcium Phosphate, Polyglactin, Catgut, Slip casting, matrix, reinforcements.

1. Introduction

A bio material should be compatible with the living body. Bioactive materials cause a constructive response subsequent to implantation regarding bone tissue creation and strengthening which thus advances recovery of the bone and its functions [1].At the present time, biomaterials of numerous types are being utilized for everything from wound dressing to tendon andl igament repair [2].Tendon and delicate tissue augmentation inserts must be sufficiently strong to withstand ordinary powers connected to them by the patient [2].

Calcium hydroxyapatite (Ca_{10} $(PO_4)_6(OH)_2$(HA)) and Tricalciumphosphate ($Ca_3(PO_4)_2$(TCP)) are perceived as ceramic materials that fundamentally reproduce the mineralogical structure of bone, material with perfect biodegradability would be supplanted by bone as it degraded[3].A biomaterial when degraded must be nontoxic and readily resorbed or excreted [4]Calcium phosphate degrades as like bones [5].Hydroxyapatite is a rising bioceramic[6]and it has good biocompatibility[7].Several materials are available for making medical instruments and implants among them hydroxyapatite is becoming the most important bio material. Nanohydroxyapatite are alike the natural apatite [8], hydroxyapatite is used to coat implants [9].Hydroxyapatite

has been used for coating 316L SS [10]. Hydroxyapatite can be utilized for bone grafting, dentistry and for blocking harmful cancer cell multiplication [11].Tri Calcium Phosphate is an appealing bone substitute material since it is an osteoconductive material. The major fact behind the usage of ßTri Calcium Phosphate is the chemical similarity as like in bone and teeth [12].By knowing the biocompatibilities of Tri Calcium Phosphate in this study it was chosen as a matrix material of the composite.

Permeable bio inserts permit improvement of bone and delicate tissues within wide pores and furthermore supply of blood for bone mineralization [13].Micropores (<10 μm) help degradation of the material, while mesopores (>10 μm) and macropores (>100 μm) have a noteworthy impact in adjustment of the underlying blood clot and resulting vascularization and reconciliation of the material in the bony tissue [1].Hence in this study porous composite material was tried.

Polyglactin and Catgut are broadly utilized as a suture material by the specialists these are accessible as absorbable sutures. Polyglactin is a copolymer of 10% lactideand90% glycolide. Catgut sutures are produced using the twisted collagen of fit ruminants and treated with chromic salt to improve assimilation period. These sutures were used in this study as reinforcing short fibers.

Composite materials give high strength to weight proportion than the generally accessible materials; thus, the focus in the improvement of composite materials for biomaterial applications has been expanded. Polymers reinforced with fibers, grafts from bone, granules of hydroxyapatite and tricalcium phosphate ceramics are some of the commonly used materials in the development of composites for biomedical applications. The prepared composites must be tested for its mechanical strength to ensure durability since it may be experienced to bending, compression and friction when functioning as implant. For ceramics indentation test is helpful to find hardness [14].

Slip casting method was used in this investigation to synthesis ceramic matrix composite; slip casting is widely used to produce ceramic components for the desired shape. To obtain the required shape, liquefied casting slip is poured into the mould and allowed to dry for the predetermined time.

2. Materials and Methods:

2.1 Materials

Tricalciumphosphate of particle size (<0.063 mm) was utilized as a matrix material of the composite. Polyglactinsutures and Catgut absorbable chromic sutures was acquired and utilized as a reinforcement fiber. Mould was created using a high density thermocol.

2.2 Methods

The methodology carried out in this study is shown as a flow chart in figure 1.

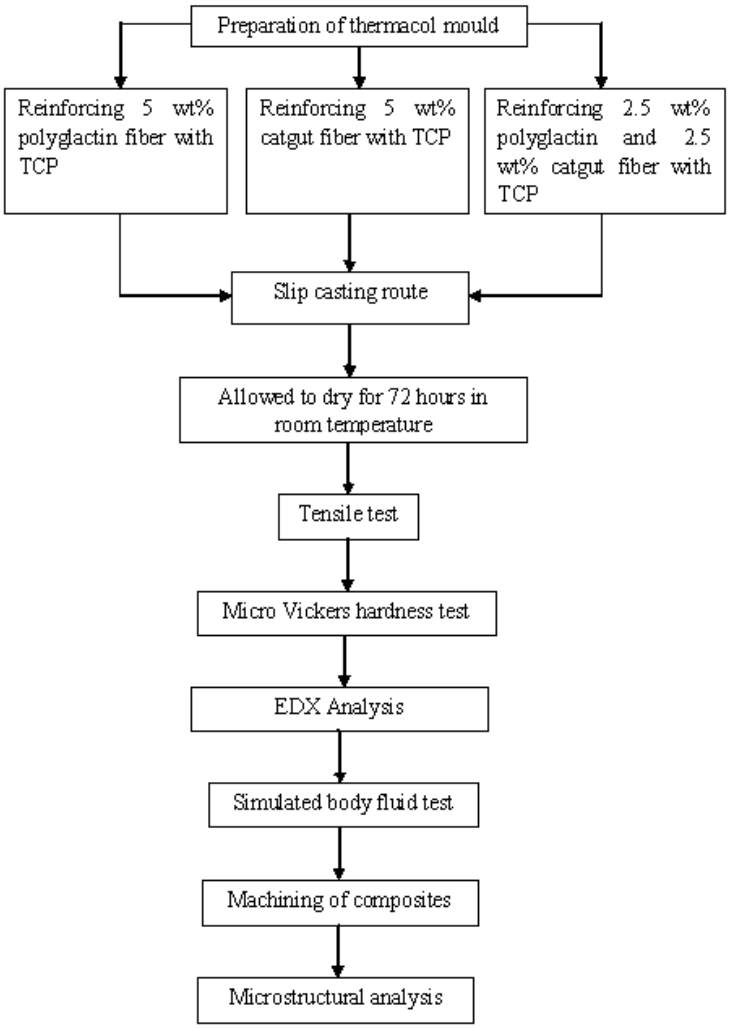

Figure 1. Flow Chart of Methodology

2.2.1 Slip Casting:

Thermocol mould in the dog bone shape was made to prepare composites with dimensional accuracies for proper clamping in the universal testing machine, Tricalcium phosphate (TCP) was blended with 5 wt % of polyglactin fibers of size 5mm long. Then it was stirred consistently for 5 minutes physically by including 10 wt% sodium silicate fluid to make a liquefied casting slip and poured in the mould, dried for 72 hours in the typical room temperature 32°C and extricated frommould. Same method was carried out for 5 wt% of catgut fiber reinforcement and for making hybrid composite of 2.5wt% of polyglactin and 2.5 wt% catgut fiber reinforcement. The filled moulds of the composites with different reinforcements are shown below in figure.2; small distortion in aspired shape of the composite was observed when extracted from the mould gentle manual filing was done to obtain the required shape to facilitate proper gripping during tensile test. The extricated specimens in dog bone shape are shown in

figure 3 (a, b&c)

Figure 2. Filled Mould of TCP and appropriate amount of fibers

2.2.2 Simulated Body Fluid (SBF) Test

The reagents used for preparing simulated body fluid are shown in table 1. The volume of the SBF required for sample testing was determined by using the formula [15]

Vs=Sa/10

Where,

Vs-Volume of Simulated Body Fluid in mm^3

Sa-apparent surface area of the sample in mm^2.

Table 1.Reagentsusedfor preparing simulated body fluid [15]

Reagents	**Amount for 1 L of SBF**
Sodium chloride	8.035 g
Sodium Bicarbonate	0.355 g
Potassium Chloride	0.225 g
Potassium Phosphate Dibasic Trihydrate	0.231 g
Magnesium Chloride Hexahydrate	0.311g
1 M Hydrochloric Acid	39 ml
Calcium Chloride	0.292 g
Sodium Sulfate	0.072 g
Tris(Hydroxymethyl) Aminomethane	6.118 g

3. Results and Discussion:

Tensile test was carried out on the three composites of 65mm gauge length and 6mm diameter to determine the ultimate tensile strength of the composite. Figure 3shows tensile specimen a) 5 wt% polyglactin, b) 5 wt% of catgut and c) 2.5 wt% of polyglactin and 2.5 wt% catgut fibers (hybrid composite).

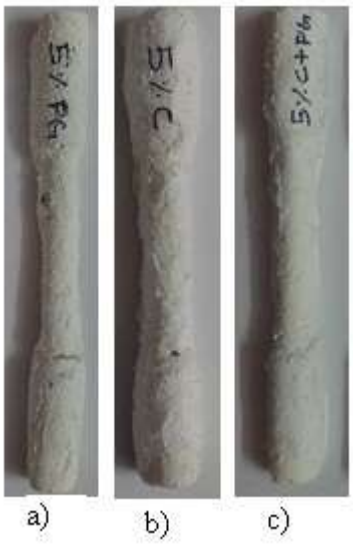

Figure 3.Tensile test specimen

Brittle fracture was observed in the failed composite specimens after tensile test, the ultimate tensile strength of the composites obtained in uniaxial loading are listed in table 2.

Table 2. Ultimate tensile strength of the composite specimens in uniaxial loading

Sample No	Composite Specimen	Ultimate tensile strength in MPa
1	TCP with 5 wt% polyglactin fiber	27
2	TCP with 5 wt% catgut fiber	26
3	TCP with 2.5 Wt% polyglactin and catgut fiber	29

Micro Vickers hardness test was carried out on the flat side of the prepared sample with 5 gfload of 15 seconds dwell time at different locations of each composite using Micro Vickers hardness tester. The hardness values are shown in table 3.The nearness of pores is a purpose behind low hardness of the composites.

Table 3.Hardnessvalueofthecomposite specimens

Sample No	Composite Specimen	Load in gf	Hardness in HV
1	TCP with 5 wt% polyglactinfiber	5	58
2	TCP with 5 wt% catgut fiber	5	59
3	TCP with 2.5 wt% polyglactin and 2.5wt% catgut fiber	5	58

EDX analysis was done using 6360(LA)instrument. The EDX result obtained from samples had high amount of calcium followed by sodium, phosphorous and significant amount of silicon. The acquisition parameter was acceleration voltage 25 kV and energy range 0-20 keV. The Ca/P ratio of the samples was 2.36 and 1.71. The (Ca+Na)/P ratio of the samples was 3.66 and 2.93. The (Ca+Na+Si)/P ratio of the samples was 5.39 and 3.62 respectively.

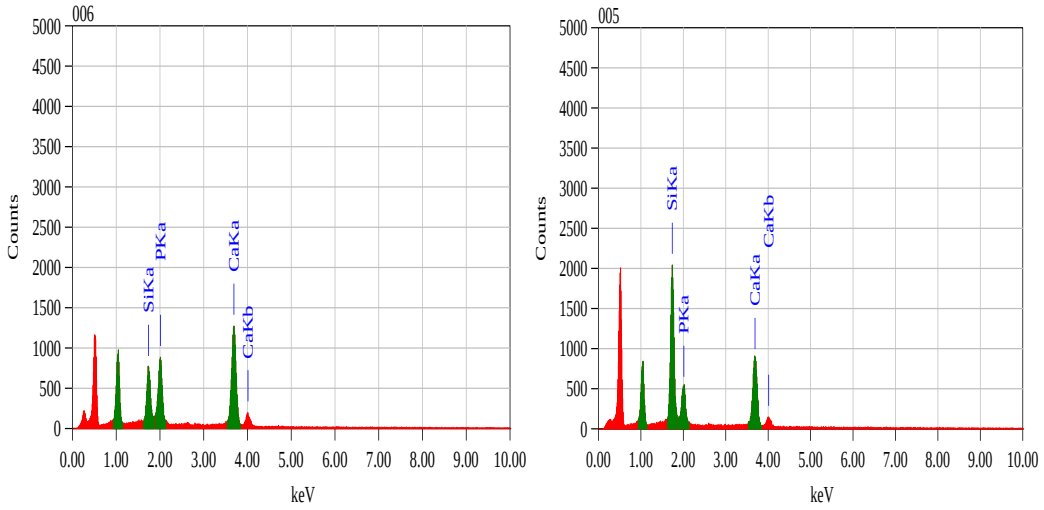

Figure 4.EDX analysis of samples

Table 3.Mass percentage of constituents obtained by EDX analysis

Element	**Mass % (Sample 1)**	**Mass % (Sample 2)**
Sodium Na	20.38	26.36
Calcium Ca	36.93	37.04
Phosphorous P	15.63	21.62
Silicon Si	27.06	14.99
Total	100	100

The prepared composites were drilled using 1mm drill bit at three different locations at center shown in figure 5 a) and at both edges as shown in figure 5 b) to study the response of material formachining. It facilitated crack free and easy drilling

Figure 5. Drilled Composites

Samples was cut and placed on the prepared wax mount and micro structural analysis was carried out at 500x using Future –Tech Corp metallurgical microscope.

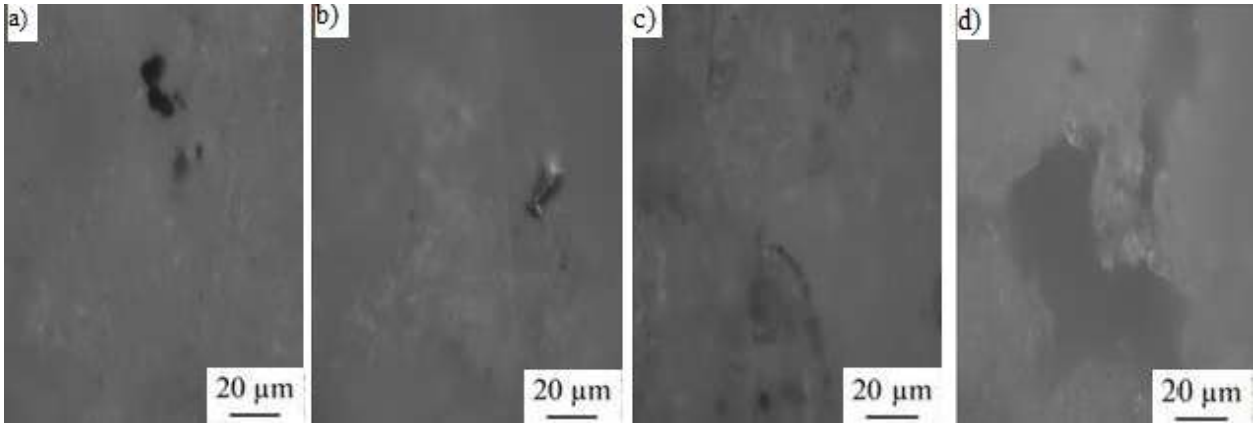

Figure 6. Microstructure of composites

Figure 6shows microstructure of composites, the micro pores of size 10µm was observed in the sample this may be due to the removal of fiber during cutting operation however the porosity increases for pure TCP sample shown in figure 6. d). the presence of porosity increased the bioactivity during invitro dissolution testing. These micro pores will allow body fluid to circulate through the sample so that dissolution of Ca^{3+} and $(Po_4)^{3-}$ ions into the surrounding fluid occur. The super saturation of these ions will enhance the formation of new bone minerals by initiating osteoblast cells.

4. Conclusion

The sodium silicate provided effective binding at room temperature drying. Tricalcium phosphate matrix reinforced with polyglactin and catgutcan satisfy the needs of orthopedic implants since it give support to pass through of body fluids and machining feasibility. Further studies can be done by varying the weight percentage of reinforcing fibers or the matrix material. Ethical in vivo studies can also be carried out for further in-depth analysis of these composites.

References:

1. Horowitz, R.A., Mazor, Z., Foitzik, C., Prasad, H., Rohrer, M. and Palti, A. β-tricalcium phosphate as bone substitute material: properties and clinical applications. Journal of Osseointegration., 2010, *2*(2), 61-68.
2. Coburn, J.C. and Pandit, A., Development of naturally-derived biomaterials and optimization of their biomechanical properties. Topics in tissue engineering., 2007, 3, 1-14.
3. Kivrak, N. and Taş, A.C., Synthesis of calcium hydroxyapatite-tricalcium phosphate (HA-TCP) composite bioceramic powders and their sintering behavior. Journal of the American Ceramic Society., 1998, 81(9), 2245-2252.
4. Ulery, B.D., Nair, L.S. and Laurencin, C.T., Biomedical applications of biodegradable polymers. Journal of polymer science Part B: polymer physics, 2011, 49(12), 832-864.
5. Engstrand, J., Persson, C. and Engqvist, H., Influence of polymer addition on the mechanical properties of a premixed calcium phosphate cement. Biomatter, 2013, 3(4), 27249.
6. Nayak, A.K., Hydroxyapatite synthesis methodologies: an overview. International Journal of ChemTech Research, 2010, 2(2), 903-907.
7. Boopathi, S. and Kumar, A.R., Synthesis and Characterization of Nanostructured Hydroxyapatite using Sol-gel Method.,International Journal of ChemTech Research,2014,6(3),2190-2192.
8. Kavitha, M., Subramanian, R., Narayanan, R. and Udhayabanu, V., Solution combustion synthesis and characterization of strontium substituted hydroxyapatite nanocrystals., Powder technology., 2014,253,129-137.
9. Brennan, S.A., Fhoghlú, C.N., Devitt, B.M., O'Mahony, F.J., Brabazon, D. and Walsh, A., Silver nanoparticles and their orthopaedic applications. Bone Joint J, 2015, 97(5), 582-589.

10. Ananth, K.P., Jose, S.P., Nathanael, A.J., Oh, T.H., Mangalaraj, D. and Ballamurugan, A.M., Biomimetic ion substituted hydroxyapatite coating on surgical grade 316L SS for implant applications., Advanced Materials Letters, 2015, 6(11), 984-989.
11. Banerjee, K., Mukherjee, M., Chaudhry, R., Pandey, N. and Thiagarajan, P., Characterization and antibacterial activity of hydroxyapatite nanoparticles. International Journal of PharmTech Research, 2016, 9(3), 452-459.
12. Sakka, S., Bouaziz, J. and Ayed, F.B., Mechanical properties of biomaterials based on calcium phosphates and bioinert oxides for applications in biomedicine. In Advances in Biomaterials Science and Biomedical Applications.InTech.2013.
13. Sepulveda, P., Binner, J.G.P., Rogero, S.O., Higa, O.Z. and Bressiani, J.C., Production of porous hydroxyapatite by the gel-casting of foams and cytotoxic evaluation. Journal of biomedical materials research, 2000, 50(1), 27-34.
14. Chicot, D., Tricoteaux, A., Lesage, J., Leriche, A., Descamps, M. and Rguiti-Constantin, E., Mechanical Properties of Porosity-Free Beta Tricalcium Phosphate ([beta]-TCP) Ceramic by Sharp and Spherical Indentations. New Journal of Glass and Ceramics,2013, 3(1), 16.
15. Marques, M.R., Loebenberg, R. and Almukainzi, M., Simulated biological fluids with possible application in dissolution testing. DissolutTechnol, 2011,18(3),15-28.

*****